17C/2004Y002

Grazing incidence small angle X-ray scattering study on pore structures of Nano-Clustering Silica

Shuichi Doi* and Naoki Awaji
Fujitsu Laboratories Ltd., 10-1, Morinosato-wakamiya, Atsugi, Kanagawa 243-0197, Japan

Introduction

Recent development of high performance CMOS devices require new interlayer dielectrics for Cu interconnects with low-k dielectric constant (k<2.3) and high elastic modulus (E=10GPa) to reduce a signal delay due to the wiring capacitances. To reduce a dielectric constant, materials which incorporate nanometer size pores have been developed. Nano-clustering silica (NCS) is one of these new materials. In such nano-porous materials, as the average size and size distribution of pores are closely relate to their mechanical rigidity and dielectric constant, the evaluation of these structural parameter become important.

Experimental

To evaluate the pore structures in NCS films on Si substrate, we carried out a grazing incidence small angle X-ray scattering (GISAXS), which is a powerful technique for structural characterization of thin layers containing nano-sized particles or pores. In the measurements, wavelength of incident x-ray beam was chosen to 0.14nm. The angle of incidence was chosen to 0.18 degrees which slightly exceeded the critical angle of NCS film to penetrate it.

Results and Discussion

Figure1 shows two-dimensional GISAXS pattern from a typical NCS film with 170nm thickness obtained by a series of detector scans. Qx and Qz are the momentum transfers parallel and perpendicular to the sample surface respectively. In Figure1, the GISAXS pattern is radially isotropic at large Q regions where the average size and shape of pores dominates the scattering intensity, while an anisotropy, like wings, can be seen at (Qx, Qz) ≈ (± 0.20,0.05). We consider that this anisotropy comes from a horizontal correlation between pores.

For the analysis, we applied the Hard-Spheres model [1], where the interference effects between particles or pores coming from their regularity are considered. To specify the pore structures of NCS films precisely, we developed out-of-plane and in-plane measurement technique, at which the detector is scanned with an offset angle from Qx/Qz=0.0 to avoid parasitic scattering such as the diffuse scattering due to the surface roughness and the tails of specular reflection. As a result of model optimization for the data, we successfully reproduced an experimental data both of out-of-plane and in-plane measurements as shown in Figure2. By assuming the spherical pore, we obtained the average diameter of 1.6nm for the vertical direction and 1.7nm for the horizontal direction respectively. We also found that pore correlation provides the pore-to-pore distance of 2.1nm and volume fraction of correlated pores is 19% in the horizontal direction, while no correlation was observed in the vertical direction.

In summary, we developed the GISAXS technique to characterize pore structures of nano-porous low-k materials. Applying this technique, the pore distributions of NCS films were obtained.

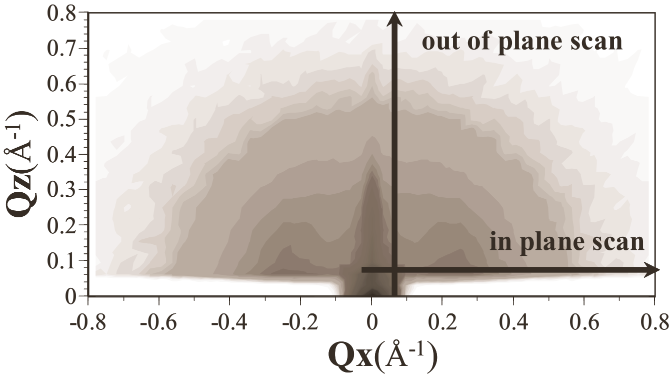

Figure1. Measured GISAXS pattern from a NCS film.

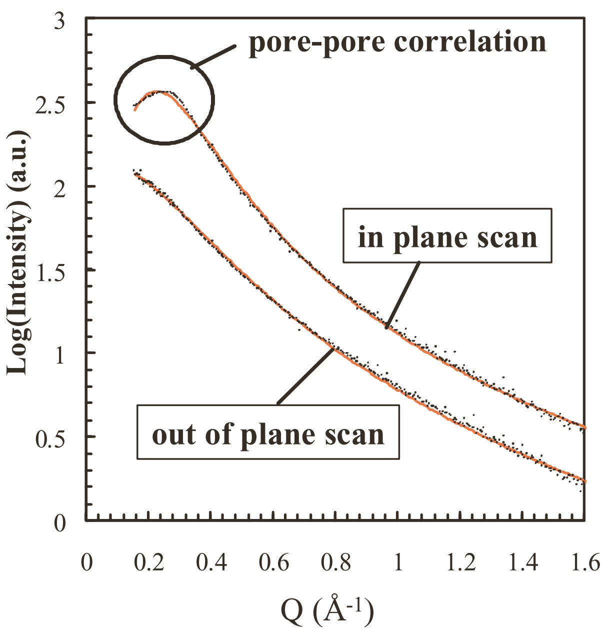

Figure2. Analysis of GISAXS data.

References

[1] J.Pedersen, J. Appl. Cryst. 27, (1994) 595.

* doi.shuuichi@jp.fujitsu.com